“Bi- Oxide / Chalcogenide Nanostructures for Energy Storage and Catalysis Applications”

Synopsis in Candidature for the Degree of

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Submitted by

Mr. Hamdan Mohsen Ali Danamah

Under the guidance of

Prof. Rajaram Sakharam Mane

School of Physical Science

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1) – Introduction

Bismuth (Bi) is brittle, lustrous and coarsely crystalline with a color of gray white, silver and pink-tinged metal. Its large variety of oxides and are BiO, Bi_2O_3, Bi_2O_4, Bi_2O_5 ,Bi_4O_7, Bi_6O_7, Bi_8O_{11} [1,2]. A few of them are stable in atmospheric pressure and room temperature. Bismuth oxide is interesting materials due to their optical and electrical properties, furthermore it exhibit high oxide ion conductivity which confer them a wide variety of potential applications. Among other properties, bismuth oxide has large energy band gap, refractive index, dielectric permittivity and photoconductivity All these characteristics make bismuth oxide suitable for several applications, such as: supercapacitor, photocatalyst and optical materials, Schottky barrier solar cells, optical coatings, and dielectric materials in metal insulator semiconductor capacitors also in the form of films or thin films we can using it important applications in gas sensor devices photovoltaic cells, microwave integrated circuits and as an oxygen conductive electrolyte in electrochemical cell. [3,4]. In general, Bi_2O_3 is a p-type semiconductor with four main crystallographic polymorphs at room temperature denoted by α, β, γ and δ [5]. Bismuth oxide thin films have been prepared by several methods, such as: Spray pyrolysis and anodic and thermal oxidation of bismuth films, reactive activated evaporation, rf sputtering, hydrothermal processes, laser ablation, etc. [6]. Bi_2O_3 a semiconductor photocatalyst with a direct band gap of [2.3-2.8] eV, can absorb the visible light region to exhibit photocatalytic behavior. However, Bi_2O_3 can be a photocatalyst when its specific surface area is high. A Bi_2O_3 powder with micro-size grains may lengthen the distance of light-generated charge carriers (electron-hole pairs) transferring to the reaction surface, lowering the energy conversion efficiency due to recombination. [7].

Electrochemical Hydrogen Evolution

The development of sustainable clean energy captures great attention of scientists in recent years due to the ever-increasing environmental pollution and energy requirements. Molecular Hydrogen (H_2) is considered as a promising candidate to meet global terawatt energy requirement in the future. Three principle steps typically occur to convert H^+ to $H_2$and to realize the hydrogen evolution reaction (HER), ($2H^+ + 2e^- \rightarrow H_2$) process in acidic media that including the adsorption of protons, electrochemical reduction, and desorption of hydrogen gas [8, 9].

Therefore, efficient and durable electrocatalysts are usually required to minimize the over potential and to reduce the free energy barriers (i.e., ΔGH) of adsorbed hydrogen species, which make the HER process more prone to proceed efficiently in both thermodynamics and kinetics.

Catalysis Application

Catalysis is the process of increasing the rate of a chemical reaction by adding a substance known as a catalyst which is not consumed in the catalyzed reaction and can continue to act repeatedly. Because of this only very small amounts of catalyst are required to alter the reaction rate in principle [10].

The production of most industrially important chemicals involves catalysis. Similarly, most biochemically significant processes are catalyzed. Research into catalysis is a major field in applied science and involves many areas of chemistry and physics, notably organometallic chemistry and the materials science. Catalysis is relevant to many aspects of environmental science [11].

In general, chemical reactions occur faster in the presence of a catalyst because the catalyst provides an alternative reaction pathway with lower activation energy than the non-catalyzed mechanism. In catalyzed mechanisms, the catalyst usually reacts to form a temporary intermediate, which regenerates the original catalyst in a cyclic process. A substance which provides a mechanism with higher activation energy does not decrease the rate because the reaction can still occur by the non-catalyzed route. The added substance which does reduce the reaction rate is not considered a catalyst but a reaction inhibitor. Catalysts may be classified as either homogeneous or heterogeneous. A homogeneous catalyst is one whose molecules are dispersed in the same phase (usually gaseous or liquid) as the reactant's molecules. A heterogeneous catalyst is one whose molecules are not in the same phase as the reactants, which are typically gases or liquids that are adsorbed in to the surface of the solid catalyst. Enzymes and other biocatalysts are often considered as a third category [12].

2)-Literature review

1- Singh *et al.* {13} they had reported that asymmetric faradaic assembly of Bi_2O_3 and MnO_2 for a high-performance hybrid electrochemical energy storage device.

In this study they explored the coupling of Bi_2O_3 negative electrode and MnO_2 positive electrode materials as an asymmetric faradaic assembly for a high-performance hybrid electrochemical energy storage device. Here they used material of Bismuth chloride sodium hydroxide and potassium permanganate and potassium hydroxide. The Bi_2O_3 nanostructure synthesized by using a simple solid-state reaction method. as well as they became to successful preparation of a Bi_2O_3 negatrode and MnO_2 positrode was performed by using facile and cost-effective chemical synthesis methods.

2- Dai *et al.* {14} reported that hierarchical electrode architectures for high energy lithium-chalcogen rechargeable batteries.

In this study they synthesized of SiO_2 product obtained by filtration and dried under vacuum before using. Synthesis of the SPC material is based on a modified reduction method. Also, they did Material characterization by the X-ray powder diffractmeter. The size and morphology of products were determined by scanning electron microscopy and transmutation electron microscopy. As well they did electrochemical evaluation cyclic voltammetry (CV) measurements were performed using a Bio-logic. They had developed hierarchical electrode architectures (SPC) by a wet synthesis method for Li-chalcogen battery applications. Excellent electrochemical result was obtained for both Li-S and Li-Se without external additives or modifications of electrode or cell configuration.

3-Arora *et al*. {15} they studied nanostructure Mos_2/$bivo_4$composites for energy storage applications.

They reported optimal synthesis and electrochemical characterization of a low-layer nanostructured MoS_2 compound along with $BiVO_4$. Compared to virgin $BiVO_4$, and a graphene / $BiVO_4$ compound, MoS_2/$BiVO_4$ nanocomposite provides great freight storage values with longer discharge times and improved stability. The results indicate that small MoS_2 molecules with an explicitly dimensioned lateral dimension provide a prominent capacitive component that helps in increasing the specific amplitude. They also discussed the optimal combination of single-layer $BiVO_4$ and low-layer nanoparticles MoS_2 using aqueous electrolyte. The obtained data shows MoS_2/ $BiVO_4$ nanocomposite that will be a candidate promising for high-energy storage applications.

4-Jin W et al. {16} they studies recent advances of porous transition metal-based nonmaterial's for electrochemical energy conversion and storage applications.

In this studies they have systematically discuss the recent achievements in the typical oxides, sulfide, phosphide and carbrides of porous transition metal-based nonmaterial for

electrochemical energy storage and conversion. Also the design was made, fabrication, and applications performance of nonmaterial's are illustrated and compared, and they have done studies of the current challenges and future prospects of porous transition metal-based nonmaterial 'sin electrochemical energy storage and conversion applications are also provided.

5- Qi, *et al.* {17} in this study they reported that progress on iron oxides and chalcogenides as anodes for sodium-ion batteries.

In this study recent progress on iron-based materials as anodes for sodium-ion batteries is summarized. Also, they provied through their studies Generally batteries can only show very limited performance at low temperature. They also arrived although iron chalcogenides show better rate performance and cycling stability than iron oxides the high operating voltage of iron chalcogenides hinders their applications

6-Sakthivel *et al.* {18} they reported synthesis and characterization of bimetallic nickel-cobalt chalcogenides ($NiCoSe_2$, $NiCo_2S_4$, and $NiCo_2O_4$) for non-enzymatic hydrogen peroxide sensor and energy storage: Electrochemical properties dependence on the metal-to-chalcogen composition. Renewable energy.

In this work, they have demonstrated the synthesis of $NiCoSe_2$, $NiCo_2S_4$ and $NiCo_2O_4$ with different structures by using a simple hydrothermal method and further used as the electrode materials for electrochemical sensor and super capacitor applications .The obtained results clearly indicate an excellent electrocatalytic activity of $NiCoSe_2$ towards the electrochemical sensing of H_2O_2 than that of $NiCo_2O_4$ and $NiCo_2S_4$. The $NiCoSe_2$ modified the electrode exhibited an ultra-low detection limit (0.03 m M) and sensitivity (8.46 m A m M^{-1} cm^{-2}) of H_2O_2 Finally, they found that the $NiCoSe_2$ is a promising electrode material for both electrochemical sensor and supercapacitor applications.

7- Dai, F *et a*l. {19} reported hierarchical electrode architectures for high energy lithium-chalcogen rechargeable batteries.

Here, they studied rationally designed hierarchical porous carbon electrode architectures with maximum micro-, meso- and macro-level porosities as the conductive framework for the lithium-chalcogen batteries. Cell level calculations suggests that the hierarchical electrode architectures have the potential to increase the specific energy to more than 350Whkg -1 ,much higher than what can be achieved by the materials and parameters. Also they developed a hierarchical electrode architectures (SPC) via a wet synthesis method for Li-chalcogen battery applications. Excellent electrochemical result was obtained for both Li-S and Li-Se without external additives or modifications of electrode or cell configuration.

8- Dai S *et al.* {20} the designed strategies in metal chalcogenides anode materials for high-performance sodium-ion battery.

In this studies they summarized recent development on the strategy of improving electrochemical sodium storage behavior for metal chalcogenide anode materials and analyzing corresponding structural fea-tures and reaction mechanisms comprehensive Generally, the common and effective methods include architecture design at nanoscale, compound with carbon material, rational and unique array structure, the control of cut-off voltage and heteroatom doping. Particular attention is placed on the design of nanocomposite arrays with various carbon matrices, which can remarkably enhance conductivity and structural stability. Finally they discussed current challenges and proposed future directions for sodium-ion battery. This benefits to further investigation and improvement of metal chalcogenide anodes for sodium-ion battery in scientific and practical applications.

9- Singh, R., *at al.* {21} reported $LiBH_4$ as solid electrolyte for Li-ion batteries with Bi_2Te_3 nanostructured anode.

The current study highlights the lithium (Li) complex hydride ions containing cluster anions. lithium borohydride ($LiBH_4$) in a very solid Li-ion battery is containing Bi_2Te_3 as an anode material. Bi_2Te_3 nanostructures were prepared using a simple, wet chemical method characterized by their crystallization drainage and electronic structure using X-ray diffraction (XRD) and electron microscopy scanning microscope (SEM). The crystalline size of nanoparticles was calculated as 19 nm and 39 nm respectively from the XRD file. The XPS study also confirms the formation of Bi_2Te_3 nanoparticles Bi_2O_3. They have observed the electrochemical performance of these nanostructures using a galvanic charge discharge curve at 0.1 °C and 0.5°C.

10- Liu X *et al.* {22} they reported nitrogen/sulfur dual-doping of reduced graphene oxide harvesting hollow $ZnSnS_3$ nano-microcubes with superior sodium storage.

In this study an ingenious two-step strategy was successfully designed to integrate small hollow microscopes from $ZnSnS_3$ and N / S-reduced graphene oxide. The resulting N/S-reduced graphene oxide $ZnSnS_3$ anode can provide high discharging capacity of 501.7 mAh g^{-1} after 100 cycles at 0.1 A g^{-1} with high Coulombic efficiency close to 100%, and a reversible capacity of 290.7 mAh g^{-1} after 500 cycles at 1.0 A g^{-1} with capacity fading of 0.06% per cycle in the potential range between 0.01V and 3.0V (vs. Na/Na +). Furthermore, the sodium storage was mechanism of the bimetallic sulfide hollow $ZnSnS_3$ nano-microcubes was revealed by in situ XRD analysis combined with exsitu XPS observation.

11- Zhou H *et al.* {23} they reported acetylacetone-directed controllable synthesis of Bi_2S_3 nanostructures with tunable morphology.

In this report, BiM bi_2S_3 microscopic and 8μm solve thermally micro flowers in the acetylasone solution were uniformly synthesized by pyrolysis of the Bi_2S_3 diethysone complex without any molds or surfactants. Bi_2S_3 microscopy was manufactured consisting of

nanorods with a diameter of 20-40 nm at 180 °C for 12 hours. In similar conditions at 240°C for 3 days, microflowers consisting of nanowires were obtained with lengths of up to several micrometers and a diameter of 20-40 nm. Here Electrochemical experiments used by Bi_2S_3 in a lithium ion battery indicated that the first discharge capacity of Bi_2S_3 microphones could reach about 148 mA/h.

12- Pandit B *et al.* {24} chemically deposited Bi_2S_3: PbS solid solution thin film as supercapacitive electrode.

It investigates the solid solution Bi_2S_3 and PbS was manufactured in the form of a thin layer by the modest method of absorption and reaction of the ionic layer (SILAR) method and is characterized by XRD, FESEM and HRTEM. This compound demonstrated excellent specific capacity of 402.4 F/g at a current density of 1mAh/cm^2 with modest charge-discharge cycles. In terms of energy storage, it showed a maximum specific power of 20.1 W/kg while accepting a specific power of 1.2 kW / kg.

3) Objectives of the study

The focus of present work *to*

(1) Synthesize and characterize Bi_2O_3 based nanostructures.

(2) Synthesize and characterize bismuth chalcogenide-based nanostructures.

(3) Investigate the influences of morphology and composition of the nanostructures on the supercapacitor and electrochemical HER performances.

(4) Study the supercapacitor performance of the fabricated electrodes.

(5) Study of Structure characterization of Bi_2o_3 by using XRD, SEM, TEM and Raman spectrometr.

(6) Study performance of the prepared electrodes in electrocatalytic hydrogen evolution reaction (HER).

(7) Study the catalytic activity of the prepared nanostructuresed electrode matireals.

(8) Application of these nanostructures in energy storage, the influences of morphologies, compositions of the nanostructures on the performance will be investigated.

(9) Compare these results with data reported previously.

(10) Design thesis accordingly based on the data available.

4) Method of the study

Spray pyrolysis method

The spray pyrolysis is an approved perspective low cost, simple, non vacuum technique [25].

Spray pyrolysis was developed to provide a large scale and low cost film growth technique that could yield similar film properties to vacuum based techniques. Spray Pyrolysis involves a thermally stimulated chemical reaction between fine droplets of different chemical species. The spray pyrolysis involves spraying a solution, usually aqueous, containing soluble salts of the constituent atoms of the desired compound onto a substrate maintained at elevated temperature. The sprayed droplet reaching the hot substrate surface undergoes pyrolytic decomposition and forms a single crystalline or a cluster of crystallites of the products. The other volatile by-products and the excess solvent escape in the vapor phase. The substrate provides the thermal energy for the thermal decomposition and subsequent recombination of the constituent species followed by sintering and recrystallization of the clusters of crystallites giving rise to a coherent film.

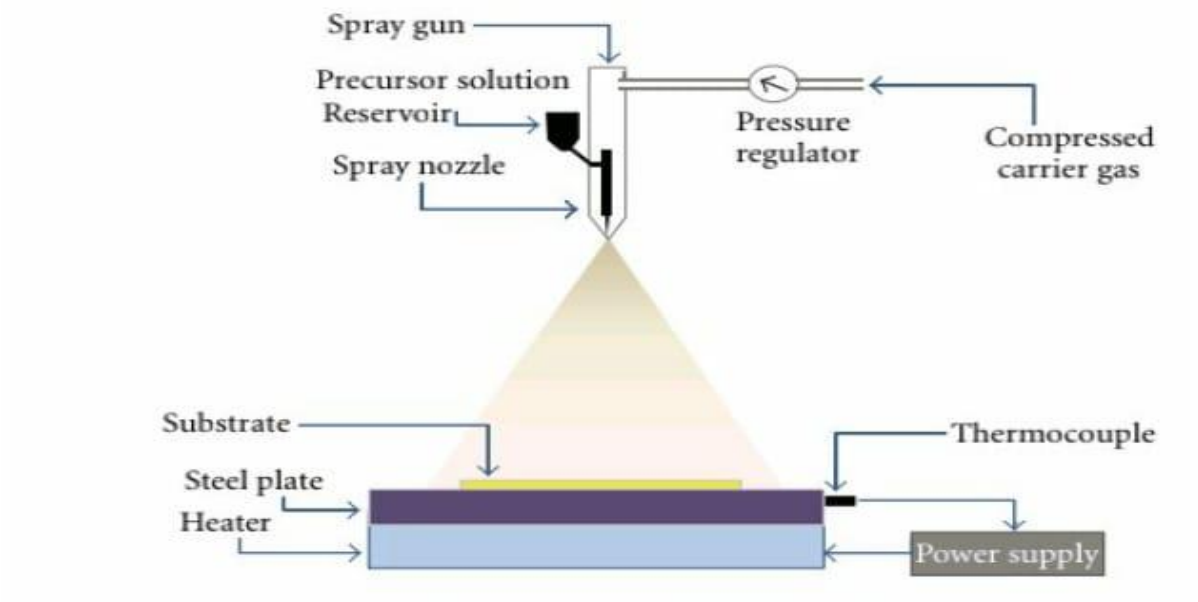

Fig. 1: Spray pyrolysis

Characterization

1- Physical characterization

a) Structural analysis

X-ray Diffraction (XRD) analysis. Diffraction method also used to differentiate the nature of crystalline materials from nanocrystalline materials and this spectroscopic technique is used to identify the atomic as well as molecular structure of crystal also used to differentiate the nature of crystalline materials from nanocrystalline materials and the structure identification is made from the x-ray diffraction pattern analysis.

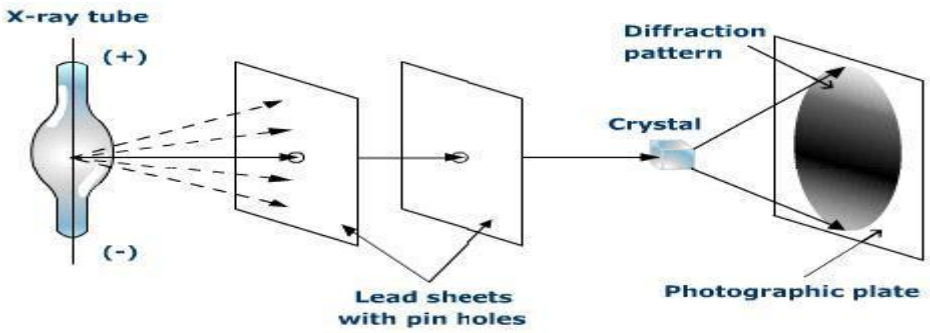

Fig. 2: Ray diagram of X-ray diffractometer

b) Surface morphology evolution

The scanning electron microscope is important types of characterization technique and it uses a focused electron beam to scan solid sample from the optical to the nanoscale range. In these types of characterization techniques, secondary electrons are emitted from the sample and it collected to create an area map of the secondary emission. The scanning electron microscopes are used to determine the external morphology (texture), chemical composition, and crystalline structure of the nonmaterial samples.

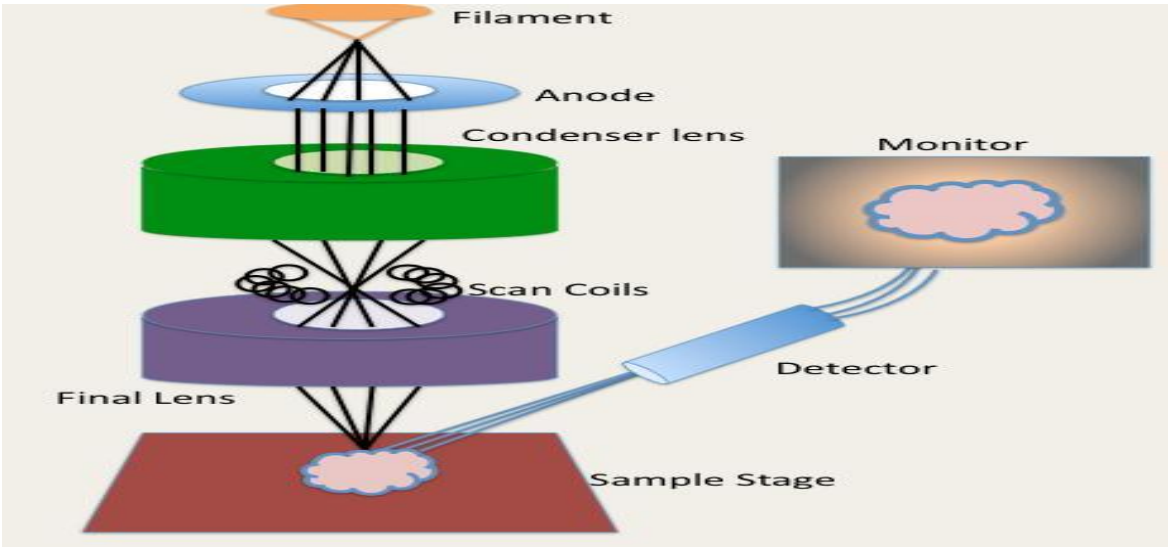

Fig. 3: Scanning electron microscopy

c) **Raman spectrometer**

Raman spectroscopy uses scattering of laser light to probe molecular structure. Of every million photons scattered, a single photon will interact with the vibrational states of a sample molecule and emit light of a different wavelength. also Raman systems can be configured to explore molecular structure as effectively as IR spectroscopy.

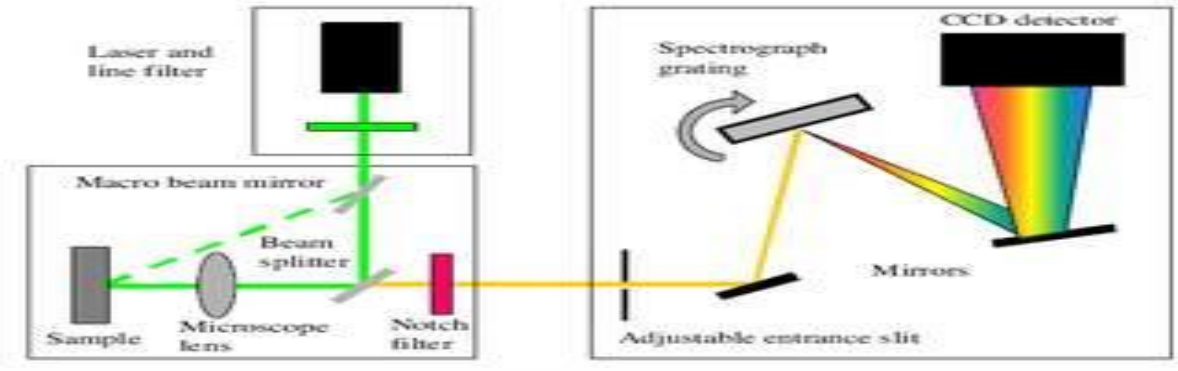

Fig. 4: Schematic diagram of a Raman Spectrometer

d) Transmission electron microscopy measurement

In this type of techniques the electrons are directly pointed towards sample. The TEM seeks to see beyond the surface and it shows sample as whole. It provides the two dimensional image and also it offers 50 million magnifications. The resolution of TEM is 0.5 Angstrom in which the beam of electron is transmitted through ultrathin specimen and it interact with the specimen as it passes and gives an image. . The TEM characterizations are used in both in physical as well as in biological sciences. Transmission electron microscope mainly was used to determine the particle size of nanoparticles. They are also used in a variety of different educational, scientific and industrial fields. They offer very high resolution and magnification.

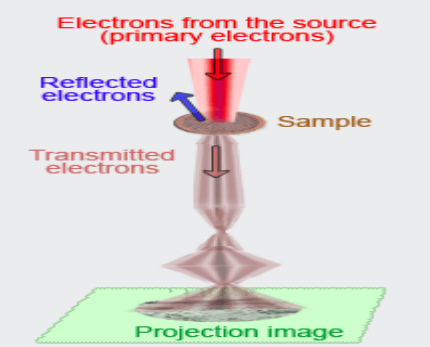

Fig. 5: Schematic diagram of transmission electron microscope.

Electrochemical measurement

Both of water spitting and supercapacitor performance are measured in a three-electrode system on an electrochemical workstation.

For electrocatalytic reaction, 4 mg of catalyst and 30 μL of Nafion solution were added into 1 mL mixed solvent of ultrapure water and absolute ethylalcohol with a volume ratio of 4:1. In order to obtain optimal electrocatalytic consequence, the mixture above was equired to form a homogeneous ink by continuous ultrasonic for more than 0.5h. Typically 5 μL of the catalyst ink is loaded onto a glassy carbon electrode (GCE).

The whole measurement process is carried out in electrolyte (eg.0.5 mH_2SO_4) using a Pt electrode as counter electrode, an Ag/AgCl (in saturated KCl solution) electrode as reference electrode, and a catalyst-modified GCE which was prepared by the steps above as working electrode.

a) **Cyclic voltammeter**

Cyclic Voltammetry (CV) is an electrochemical technique which measures the current that develops in an electrochemical cell under conditions where voltage is in excess of that predicted by the Nernst equation. Cyclic Voltammetry can be used to study qualitative information about electrochemical processes under various conditions, such as the presence of intermediates in oxidation-reduction reactions, the

reversibility of a reaction. CV can also be used to determine the electron stoichiometry of a system, the diffusion coefficient of an analyte, and the formal reduction potential, which can be used as an identification tool.

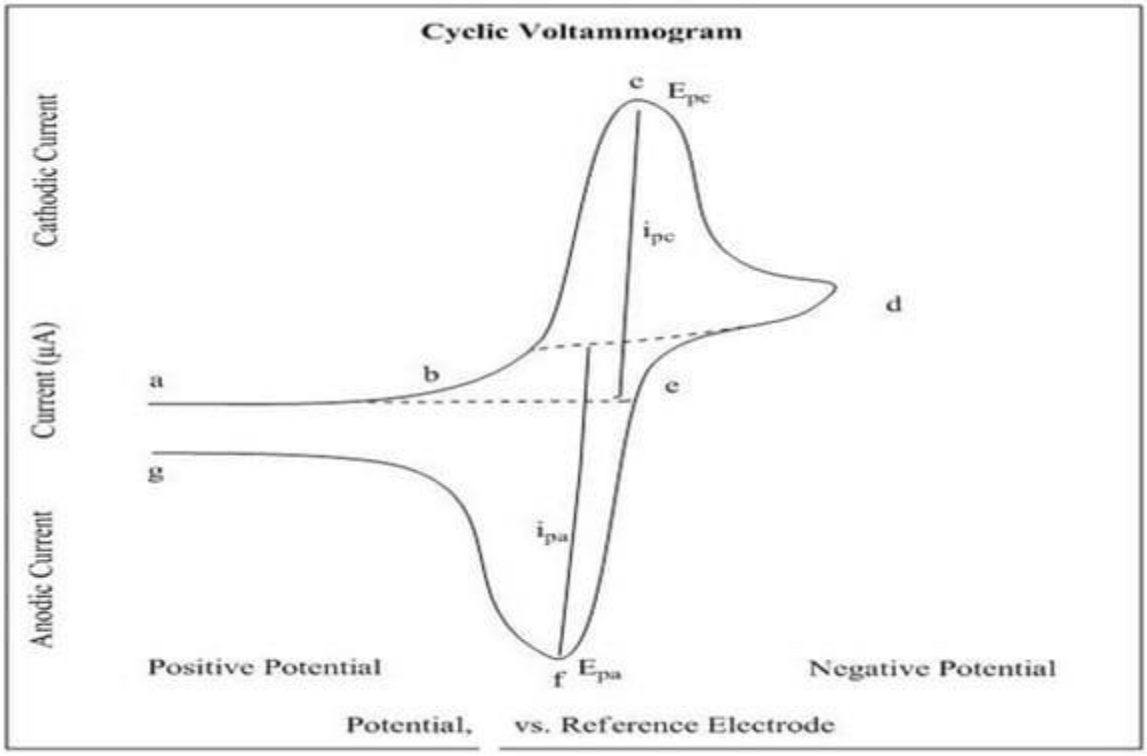

Fig. 6: Cyclic voltammetry

b) X-ray photoelectron spectroscopy (XPS)

Is a surface-sensitive quantitative spectroscopic technique that measures the elemental composition at the parts per thousand range, empirical formula, chemical state and electronic state of the elements that exist within a material. Put more simply, XPS is a useful measurement technique because it not only shows what elements are within a film but also what other elements they are bonded to.

c) Electrochemical impedance spectroscopy (EIS)

Is an electrochemical technique to measure the impedance of a system in dependence of the AC potentials frequency. As well as is a powerful tool to study chemical and physical processes in solutions as well as in solids. Electrochemical impedance spectroscopy (EIS) is a powerful nondestructive technique that can act as a beneficial addition to the current techniques for studying microbial fuel cells (MFCs).

d) Supercapacitor performance

For supercapacitor performance, very similar to HER, an Hg/HgO electrode is used as reference electrode and a Pt electrode as counter electrode, while a piece of nickel foam coated with a certain amount of as-synthesized products is acted as the working electrode. In supercapacitor performance measurement, the CV curves are performed by sweeping potential. Versus Hg/HgO electrode under various sweep rates.

Galvanostatic charge–discharge (GCD) curve is another intuitionistic approach to obtain specific capacitance. The GCD curves were performed at different current densities.

e) Electrochemical hydrogen evolution reaction (HER)

The polarization curves are obtained under the condition of different sweeping potential. The chronoamperometry j–tcurve is measured in a constant voltage. The Electrochemical impedance spectroscopy is obtained in the same condition at particular overpotential, frequency range and an amplitude.

f) Oxygen evolution reaction (OER)

Is a limiting reaction in the process of generating molecular oxygen through chemical reaction, such as the oxidation of water during oxygenic photosynthesis, electrolysis of water into oxygen and hydrogen. Developing improved catalysts for the OER is the key to the advancement of a number of renewable energy technologies, involving solar fuels production and metal–air batteries. Oxygen evolution from water is effected by oxygenic photosynthesis, electrolysis of water, and thermal decomposition of various oxides.

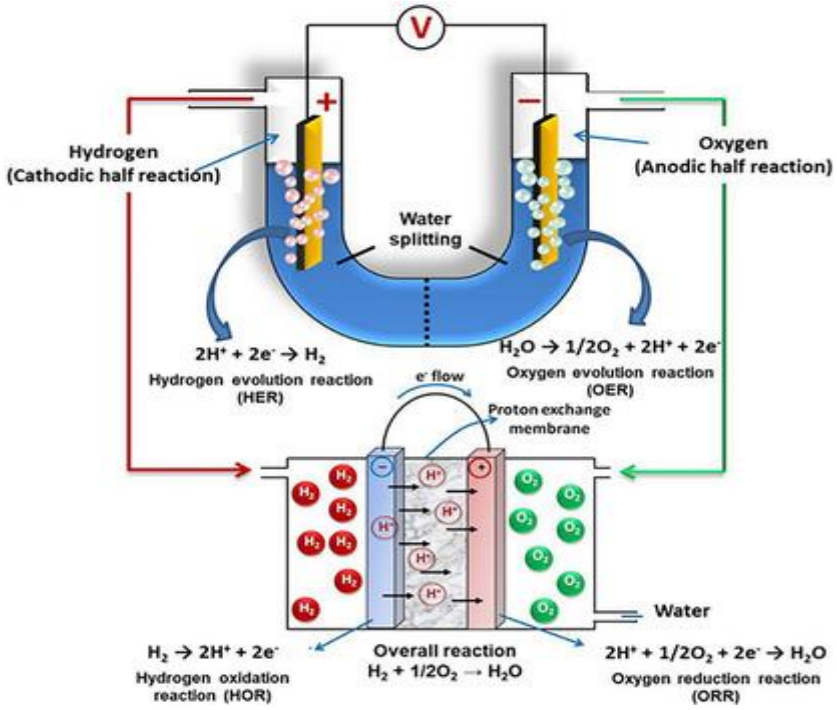

Fig. 7: Operation of water electrolysis unit.

g) Galvanostatic charge- discharge

As an important measure in supercapacitor, the charge-discharge curve gives all information about electrode material including specific capacitance, stability, power and energy densities etc. The current density remains constant in charge- discharge curve with constant potential window and it can be changed for obtaining different values of the

discharge time between the two potentials. In charge process the curve may begin from zero time or any value in the range of potential window and it increases linearly up to the desired potential. Then, it reveres its direction to come back to the original value within a certain time.

5) Problem undertaken

The wide applications of nanostructured are based on the progress in the synthesis of new nanostructured materials of different sizes and new morphologies. Therefore, further developing new materials with low overpotential and better performance relative to existing electrode materials is the most important technique to overcome these challenges. The best materials that can be used as an electrode in Store energy, as well as supercapacitor, are Bismuth oxides / chalcogenide materials due to their wide potential window, highly redox reactions, distinct oxidation states, high conductivity, remarkably low overpotential, good thermal stability, long cycle life, and high rate capability as well as it is an environment friend. In addition, they were also found to be a versatile backbone to composite bismuth oxides based nanostructures of known value for overpotential. Ternary bismuth oxides involving offer great promise in the future of electrolysis materials as they can contribute to the good overpotential of the system by an added chalcogenide while the unique combination enable better chemical stability.

My plan to

Chapter one Introduction.

Chapter two Experimental Techniques.

Chapter three Layered Oxychalcogenides Bi_2O_3CuCh (Ch = S, Se, Te) and $Bi_2YO_4Cu_2Se_2$.

Chapter four Structures and Thermoelectric Properties of p-type Oxychalcogenide

Bi_2O_2Ch (Ch = Te, Se) Materials.

Chapter 6 Conclusions .

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Research scholar

Hamdan Mohsen Ali Danamah

26/12/2021

Research Guide

Prof.: R .S. Mane

Professor
School of Physical Sciences
S.R.T.M.University,Nanded.

S.R.T.M.U. Nanded